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Experimental Study of Low Amplitude, Long-Duration Mechanical Loading of Reactive Materials

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Experimental Study of Low Amplitude, Long-Duration Mechanical Loading of Reactive Materials.

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ABSTRACT

Studies of the low amplitude, long-duration mechanical loading of reactive materials rely very heavily on the experimental data in general and in particular on the data obtained from gauges placed within the experimental test sample to measure accurately the local changes of parameters of the investigated material. For a complete description of these changes taking place in a dynamically loaded material one would like to know both the spatial and the temporal resolution of pressure, temperature, volume, wave and mass velocity. However, temperature and volume are not easily attainable. Therefore, most of the in-situ work is limited to measurements of pressure and both wave and mass velocities.

Various types of these gauges will be discussed and their records will be illustrated. Some of these gauges have limitations but are better suited for particular applications than others. These aspects will also be discussed. Main limitation of most in-situ gauges is that they are built for one-dimensional application. However, some work is being done to develop two-dimensional gauges. This work will also be briefly discussed.

While these experiments are necessary to validate theoretical models of the phenomenon, they can also provide sufficient amount of data to yield complete information on material characteristics such as its equation of state (EOS), its phase change under certain loads and its sensitivity to shock loading. Processing of these data to get important information on the behavior of both reactive and non-reactive materials will also be demonstrated.

INTRODUCTION

Mechanical loading considered here is of dynamic nature provided on a material by a shock wave. A shock wave is a phenomenon whereby a material through which it propagates will experience a sudden and discontinuous change of state. When materials are subjected to such dynamic loading they behave differently and sometimes undergo a dramatic change of state due to chemical reaction or even physical phase transformation. Proper knowledge and understanding of these changes is required to properly characterize these materials by their appropriate equations of state.

For many years the only information readily available was the shock velocity measurements, which in some cases were not very sensitive to variations of other more important variables, such as pressure and particle velocity. This is especially true when the flow behind the shock wave is not steady but is rather highly time dependent due to some new processes caused by the shock wave. Explosive materials are the outstanding example of a chemical process triggered by a shock wave. Physical phase change of some materials is another example. Parameters in such unsteady reaction media can become rather complicated and require more sophisticated and accurate measurements.

The desired accuracy and resolution of experimental measurements depends very heavily on actual values of the parameters as well as on the rates of change of these values during the loading process. The diagnostic tools chosen for these measurements must be capable of following these changes and faithfully recording the new attained values. During the shock wave loading process the major parameters which undergo a drastic change are density - ρ, pressure - P, temperature - T and particle velocity - u_p. Of these pressure and particle velocity are the most desired ones for theoretical analysis since they appear in the two basic conservation equations and can be used to properly identify the changes taking place in the material under shock loading.

In inert materials pressure and particle velocity data together with the shock velocity information provide the basic equation of state data yielding U_S - u_p and P - u_p, i.e., shock velocity - particle velocity and pressure - particle velocity relationships respectively [1].

In reactive media the time resolved histories of these two parameters can further lead to calculations of λ the time resolved reaction parameter and even $\partial\lambda/\partial\tau$ time resolved reaction rate parameter[2]. These are the two characteristic values of a reactive medium in a non-steady regime where a slow burning process gradually builds up and develops into a violent detonative explosion.

The purpose of this communication is to extend our earlier publication on the subject[3]. The intent is to review some of the existing experimental techniques available to us for studies of such high rate processes, to describe their basic principles and to point out recent modifications and improvements to these techniques that provide better resolution and better accuracy. This review does not cover all the techniques in shock wave research but to point out some of them which are most common and which are successfully used in our laboratory.

EXPERIMENTAL TECHNIQUES

Some experimental techniques use optical equipment to visualize the event from a distance without making direct physical contact with the material that was brought into motion by the shock wave. If the material is transparent, as most of the gaseous media are, then one can observe the changes occurring within that medium i.e., inside of its boundaries or containers. However, most of the materials of interest are opaque. Here optical techniques can only observe those changes, which are translated to the outside shell of the experiment, i.e., either to the surface of the investigated material itself or to the wall of its original container. From these observations one must then make proper inference of what really took place within the sample. This is not always easy to make because the observation is usually made on a free surface boundary, which cannot sustain the original dynamic loading.

In contrast to the diagnostic schemes which do not interfere with the flow, there are the thin film gauges embedded into the sample, and sensing time resolved changes, which take place within the moving medium. Such gauges provide more direct information on the histories of the desired parameters and therefore can resolve smaller local variations. The usual concern here is to make the gauges small but sufficiently strong to survive the hostile enviromnental of extreme pressures and temperatures and still accurately respond to the fast changes taking place at the location of the gauge.

The dynamic loading of material can be accomplished in many different ways. However, in order to properly describe the behaviour of the material under such unusual environment, one must have an accurate account of that environment and have a good control of loading conditions.

One of the ways to control the shock strength or the characteristic features of impact loading is by the use of a gas or a powder breach gun to accelerate a projectile of known material with a known mass to provide a flat impact with the target. The gun in our facility at the Lawrence Livermore National Laboratory has a bore of 101 mm and is capable of reaching projectile velocities of 0.2 - 2.7 mm/μs. Depending on the materials used for such experiments one can produce impact pressures in excess of 1 Mb. A schematic representation of such a gun experiment is shown in Fig. 1 where the projectile with the proper flyer plate is shown just after leaving the gun barrel but before impacting the target assembly.

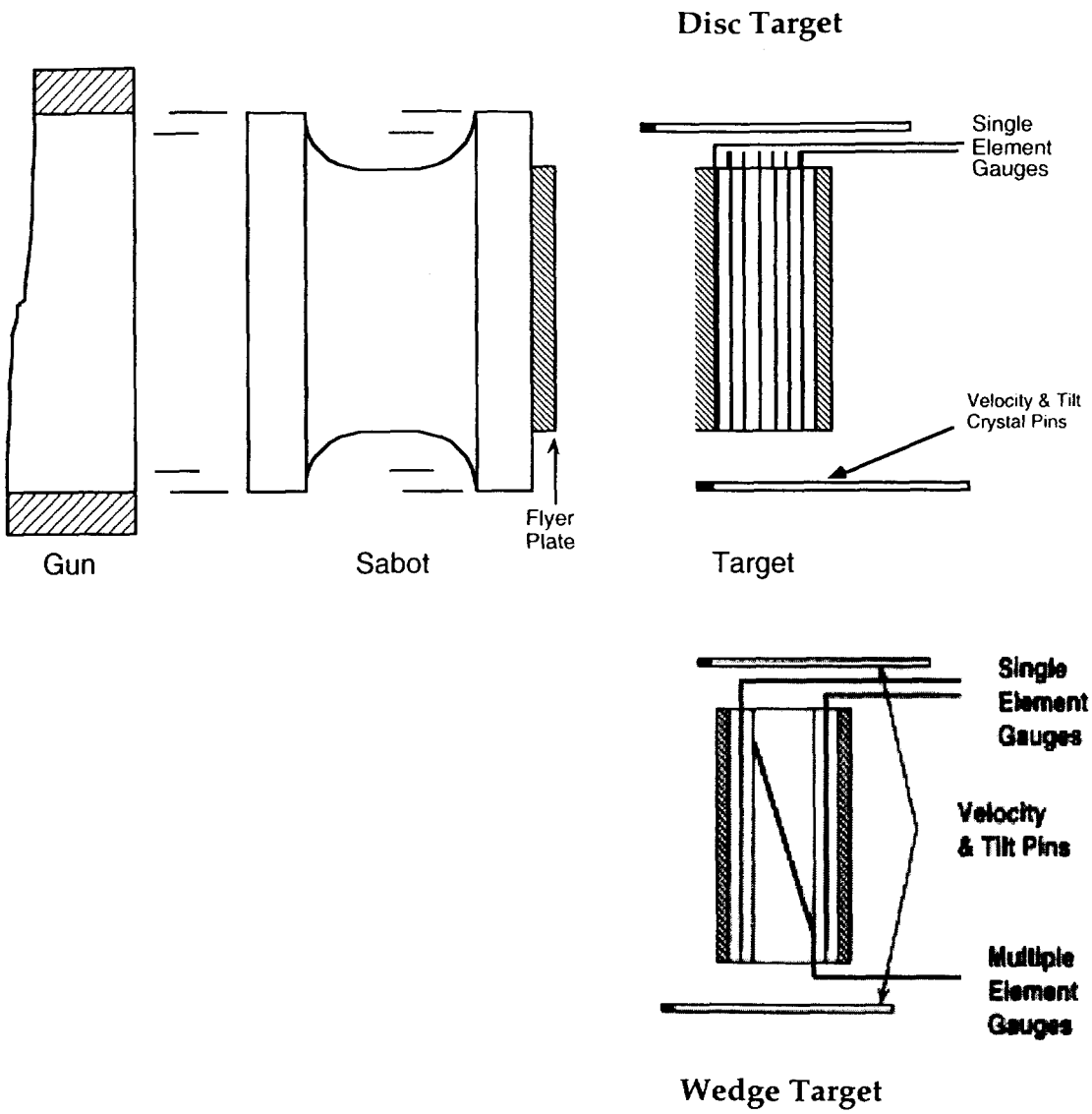

Figure 1. Schematic of gun experiments

The target can be of any form to accommodate any type of gauges and/or pins, which are best suited for measurement of the desired parameter. Usually it consists of several layers of the investigated sample with gauges embedded between them. Such multiple-gauge records were found to be quite adequate for some applications. However, it was also quite apparent that separate discs with gauge packages between them cause undesirable disturbance in the flow due to material impedance mismatch of the materials. They also cause significant interruption of the reaction process due to the inert nature of the armor materials. To overcome these problems several gauge elements were combined into one single gauge package and placed at an angle to the oncoming flow as illustrated in the figure. Each gauge element is then at a different depth in the flow and this depth can easily be controlled by the separation of the elements and by the angle of placement. This then not only eliminates the multiple sources for flow disturbances but also enhances our measuring capability by giving us an unlimited control of depth in positioning the gauge and at the same time increases the number of measurements on a single experiment. Also included on the target are several crystal pins of different length to measure the velocity of the impacting flyer plate in addition to six flush mounted crystal pins, which are placed around the periphery of the target, whose sole purpose is to measure the amount of tilt during the impact.

Different types of gauges are used to measure different parameters. As mentioned before, the most important parameters in analyzing shock wave processes are shock velocity, particle velocity and pressure. Temperature is another parameter which is of great interest but as yet no real progress has been made to develop a proper gauge to respond and follow fast changes and high values of temperature brought about by a shock loading process.

In the following sections only a few basic gauges are described, leaving the readers to expand their further knowledge on the subject on their own.

TIME OF ARRIVAL CRYSTAL PINS

To measure the shock velocity one can use a row of crystal pins placed on the surface of the wedge sample[4] as shown in Fig. 2. As the shock wave propagates along the sample it will encounter one pin after another providing the time of arrival of this pin's location. These data can be plotted on a distance time plot as illustrated in Fig. 2 where the slope of the resulting line, ($\Delta x/\Delta t$), will provide the value of the desired parameter.

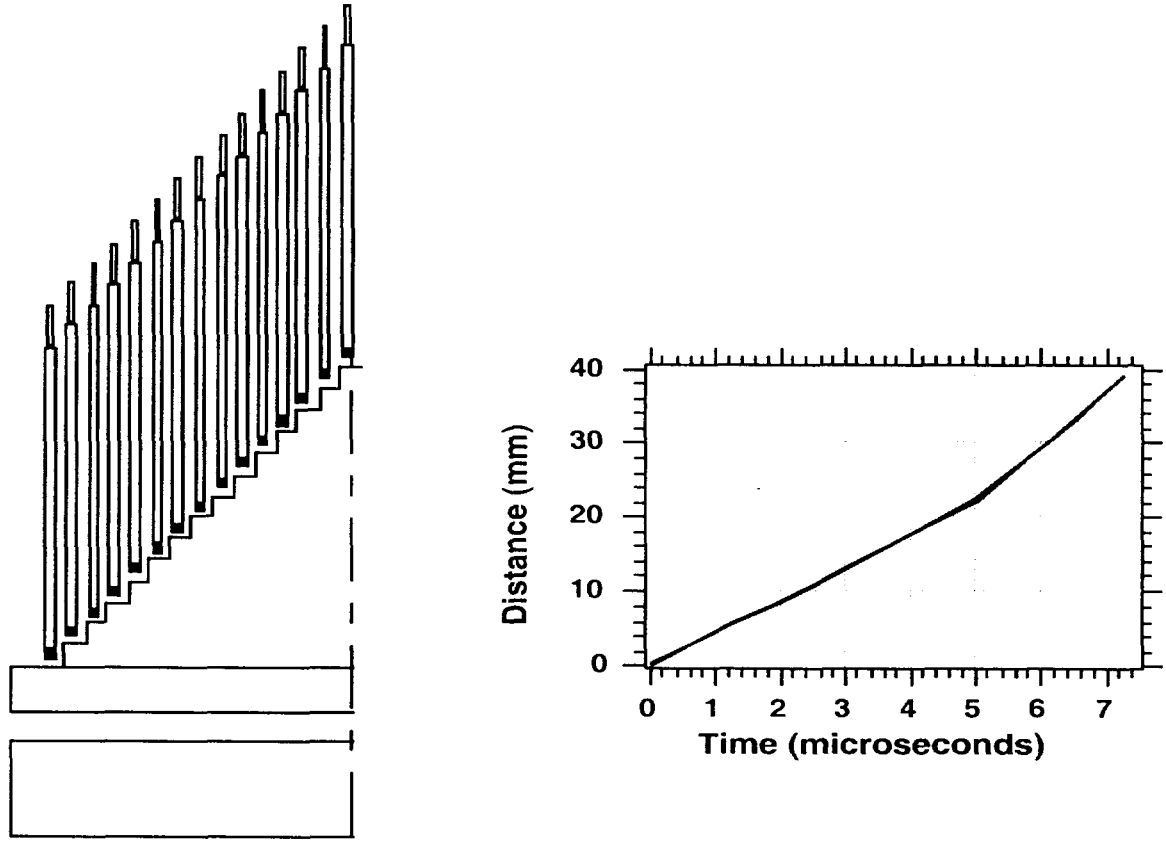

Figure 2. Experiment illustrating pin diagnostics and x-t diagram from experiment

If the wedge sample is inert the shock velocity should remain fairly constant. However, if the sample is made of reactive material the slope on the data plane may show a change to a higher velocity. This would then mean that the sample detonated and the distance at which this event occurred represents the transition distance to detonation. This distance to detonation depends on the strength of the loading shock wave i.e., its pressure. The stronger the shock wave, the shorter

the distance. This loading pressure - distance relationship is unique for each explosive and, therefore, serves as a measure of explosive sensitivity to impact. Such a sensitivity plot was originally proposed by Ramsay and Popalato[5] and is better known as the "Pop Plot". A representative of such a plot for several explosives is shown in Fig. 3. The closer the line is to the origin of the plane the more sensitive is the explosive.

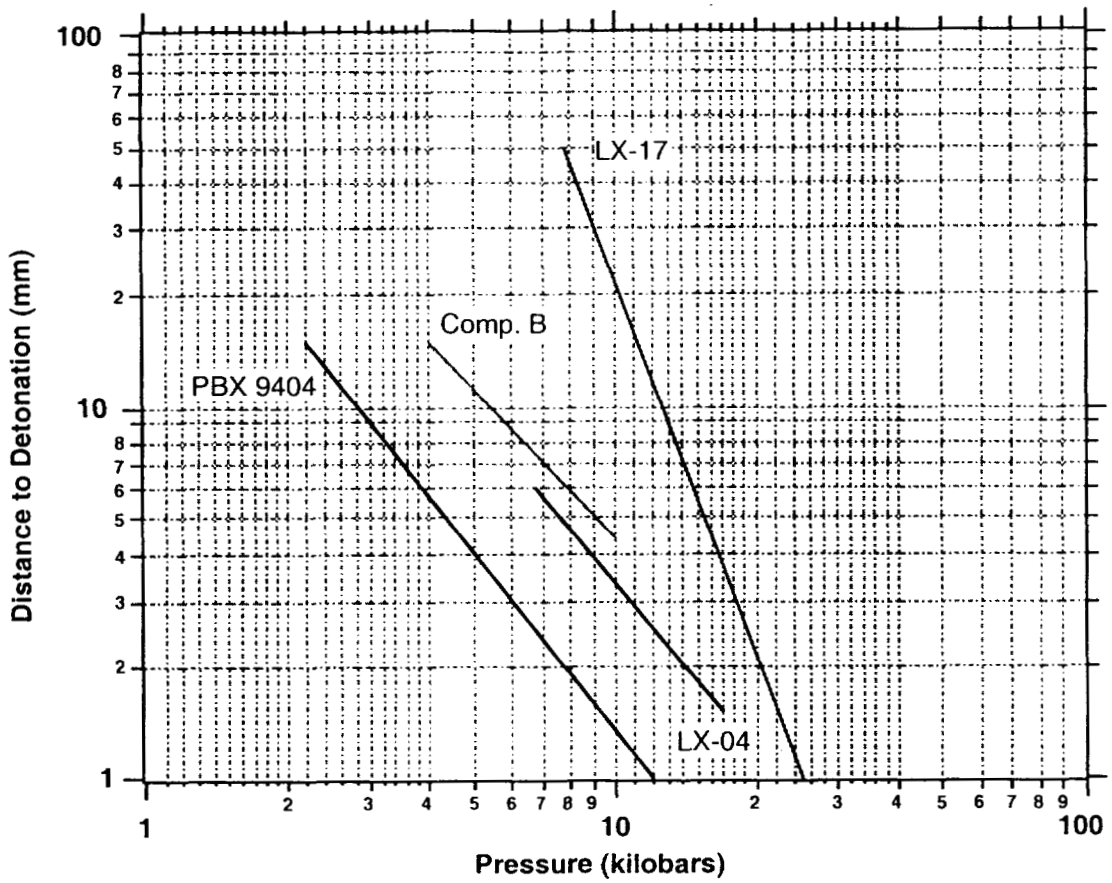

Figure 3. Pop plots of various explosives

The particle velocity gauge operates on an electro-magnetic principle. It is not new but due to marginal quality of early results, was dormant for a long time and had very limited application. Original development of the gauge was done by Zavoiski in 1948 according to Dremin and his coworkers at the Institute of Chemical Physics in Moscow. Many years later Dremin et al.[6] have reactivated this technique which then found its way to other laboratories in the USA. Here Edwards et al.[7] and Hayes and Fritz[8] further developed the systems and improved its reproducibility and accuracy.

The thin foil particle velocity gauge was brought close to perfection and is now being used regularly at our Laboratory as a standard diagnostic feature in the 4 inch bore gas gun experiments. Its shape and form are constantly being modified to provide the experimenter with more sophisticated and more accurate data.

There are two types of particle velocity probes that are used at the Lawrence Livermore National Laboratory, namely the Lorentz gauge, which has the leads come out on the side and the Faraday gauge, where the leads come out at the back. The Lorentz gauge is shown in Fig. 4. Each type is a transducer that converts displacement rate to an equivalent electrical voltage. The reason

for distinguishing between the two types of transducers is that the Faraday gauge is a low impedance device capable of operating in conducting detonation products without protective insulation, while the Lorentz gauge must be armored with a protective plastic to maintain its integrity in a hostile environment. This additional plastic covering adds to the gauge thickness. This in turn causes the system response to decrease, which limits the time resolution to fractions of microseconds rather than a few nanoseconds.

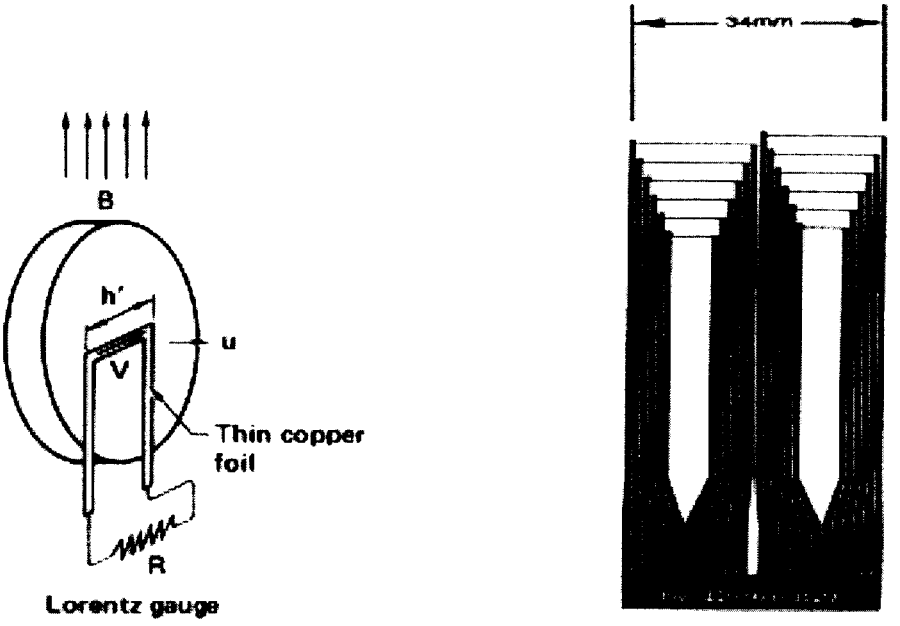

Figure 4. Exampls of a single and a multiple emv particle gauge

At present, the Lorentz single and multiple gauges are shown in Fig. 4 , one dimensional, thin foil, hydrodynamic particle velocity sensors. They may be made of copper or aluminum, depending on the application. Note that copper, while not as good in terms of shock impedance match, survives better than Aluminum in the severe environment of the detonation products. Also shown in this figure is the simple expression, which was used to reduce the data. Here E(t) is the electrical signal in volts, B is the magnetic field in kG, h is the length in cm of the gauge element and $u_p(t)$ is the resulting particle velocity in mm/μs.

Particle velocity history data are very useful in validating theoretical models. Multiple gauge experiments reveal the information on the build-up to detonation as well as information on the release wave behind the detonation front. A composite diagram showing a typical multiple gauge experiment with particle velocity histories is shown in Fig. 5. The flow is generated by the impact of the flyer plate at the front surface of the shot assembly. The moment of impact is taken as the zero time at which two shock waves are generated: one propagating into the sample with the embedded multiple element gauge and one traveling back into the flyer plate.

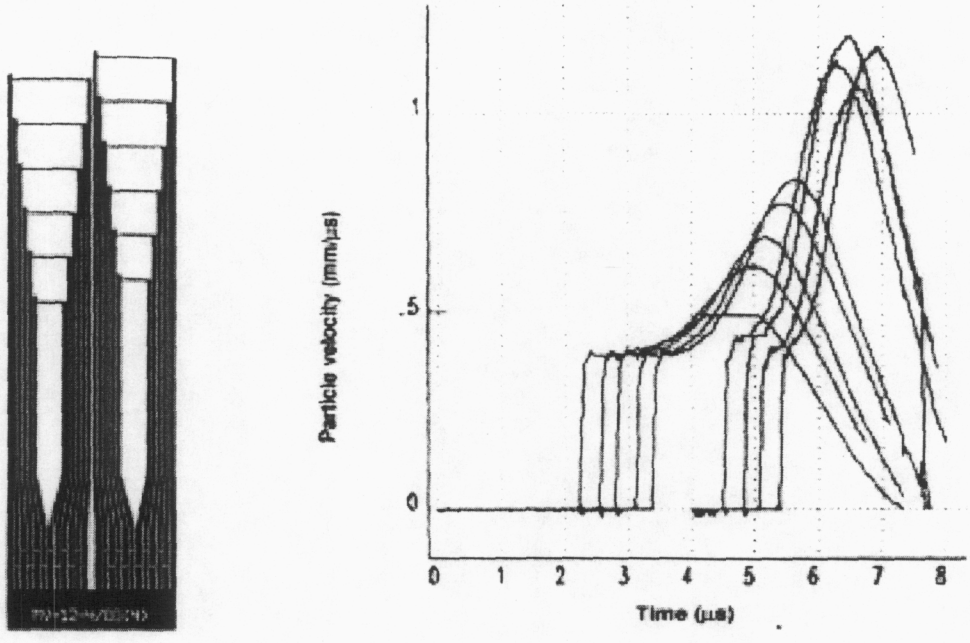

Figure 5. Multiple particle gauge and records of LX-14 experiment

The gauge package shown in Fig. 5 contains twelve particle velocity gauge elements placed into a LX-14 explosive (95.5/4.5 wt %; HMX/ Estane). As the shock travels across the gauge, the gauge elements are set in motion crossing the magnetic field and thereby generating a current, which then is appropriately recorded. Taking into account the strength of the magnetic field and the length of the sensing element, the signal is then converted into the units of particle velocity. These measured particle velocity traces are shown in the figure. The records clearly show a continuous rise of particle velocity indicating that reaction is taking place and the process is well on its way to turnover to detonation.

PRESSURE GAUGES

Manganin was first used as a pressure transducer in a hydrostatic apparatus by Bridgeman[9]. More than a decade later manganin wires were used first as dynamic stress transducers in inert materials and then as a gauge in reactive materials to study shock initiation and Taylor wave profiles in various explosives[10-12]. Weingart et al.[13] and Erickson et al.[14] devised a low impedance gauge etched from foil and insulated with polytetrafluoroethylene (PTFE) for use in making in situ measurements in reacting and detonating high explosives. This use has been described in our recent publications[15-16].

Manganin is well suited for use as a pressure transducer because it has a reasonably high coefficient of resistance change with pressure, the coefficient is positive, and its response is nearly linear. Of even greater importance is the fact that it has a very low coefficient of resistance in response to changes in temperature which occur due to shock heating[17-18]. It does, however, exhibit a small amount of hysteresis[19-21], e.g., once subjected to a given shock pressure it undergoes a permanent change in resistance.

Our manganin gauges are fabricated at LLNL using annealed, shunt grade manganin foil of 25.4 μm thickness. The foil is composed of 85.90% (wt%) copper, 9.5% manganese, 4% nickel, 0.5% iron, and 0.1% silica. The transducers are etched from the foils using standard techniques and the electrical leads are plated with 8 to 10 μm of copper to reduce the lead resistance. Since the gauges have a low resistance it is necessary to make a four-terminal resistance measurement.

Our gauge configuration is a single-ended one in which all four leads emerge from one side. This configuration is illustrated in Fig. 6. It offers greater flexibility in gauge placement within a target assembly. The active element of the single-ended gauge is 2.0 x 0.7 x 0.025 mm; its nominal

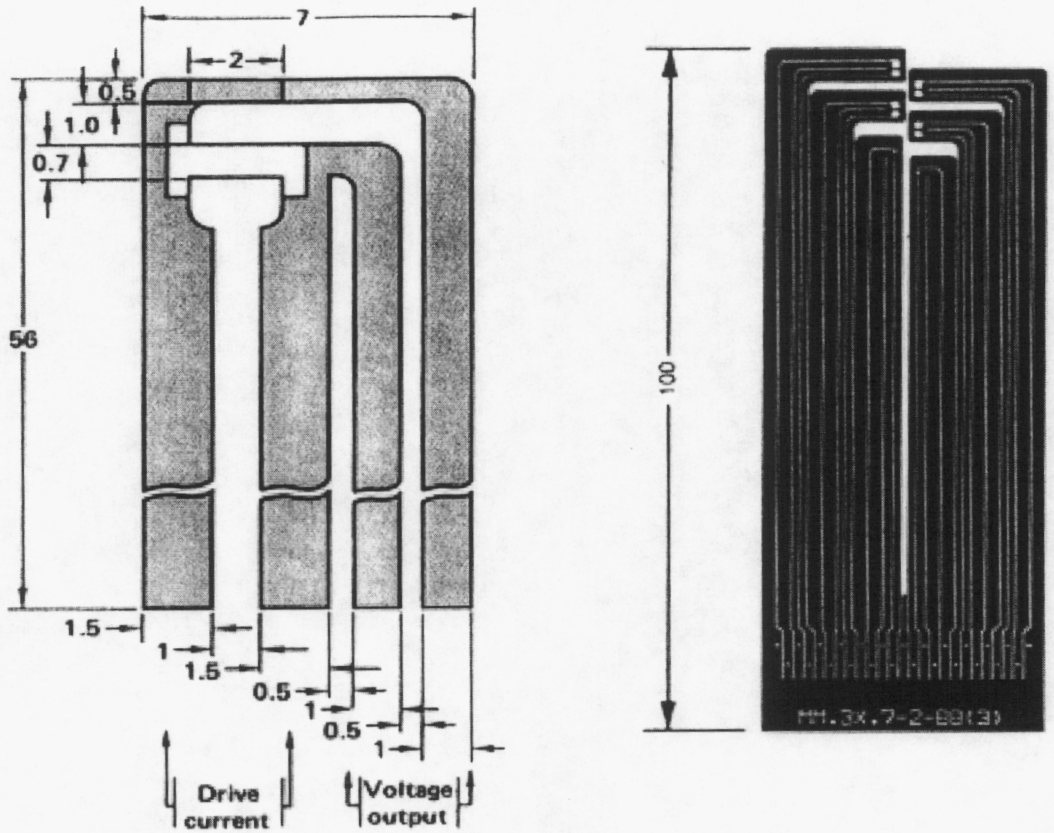

Figure 6 Sketches of the four lead single and the six element multiple manganin pressure foil gauge

The multiple element version is also shown in this figure, which is placed at an inclined surface. This gauge shows six gauge elements separated from each other by 3 mm. As in the case of the particle velocity gauge this spacing can be varied and we have made gauges with 2, 5, and 8 mm intervals as well. The gauge elements are smaller than those in a single element gauge but they are kept at the same aspect ratio to keep the same nominal resistance R_0. Their size is 0.7 x 0.3 x 0.025 mm.

Etched gauges are bonded into "gauge stations" where gauges are sandwiched between layers of PTFE dielectric. The bonding agent is Fluoroethylene Propylene (FEP), and the bonding is achieved by placing the sandwich in an evacuated fixture and inserting into a press preheated to the melt temperature of the FEP. Details of construction have been reported by Erickson et al.[14].

During the experiment constant current (usually 50 A) is supplied to the gauge prior to shock arrival, producing a resultant ambient voltage, V_O. The arrival of the shock wave produces an additional voltage ΔV. To enhance the accuracy of the measurement we use a modified bridge circuit to offset the ambient voltage as described by Vantine et al.[22]. The current in the gauge is measured and the fractional resistance change is related to measured values of V_0, ΔV, I, and R_O as:

$$\frac{\Delta R}{R_o} = \frac{V_o + \Delta V}{IR_o} - 1 \qquad (1)$$

Here R_0 is the initial resistance of the gauge measured to $\pm$0.01% accuracy, V_0 is the ambient voltage, I is the current through the gauge, and ΔV is the change in voltage due to the pressure. Each gauge responds to pressure by changing its resistance. Since the current flowing through the gauge element is constant any change in resistance is reflected in a change of the voltage signal. By monitoring both current input and voltage output signals and applying a proper conversion polynomial[21], one can get an accurate account of the pressure change as seen by each individual gauge element.

The results of the multiple manganin gauge experiment are illustrated in Fig. 7 where the gauge was placed in an explosive target of LX-14. Such gauges are very valuable for measuring pressure along a Lagrangian space coordinate where the pressure builds up and at some point may even turn over into the detonation wave. In the case at hand the turnover did not happen within the extent of of our measured length of the target.

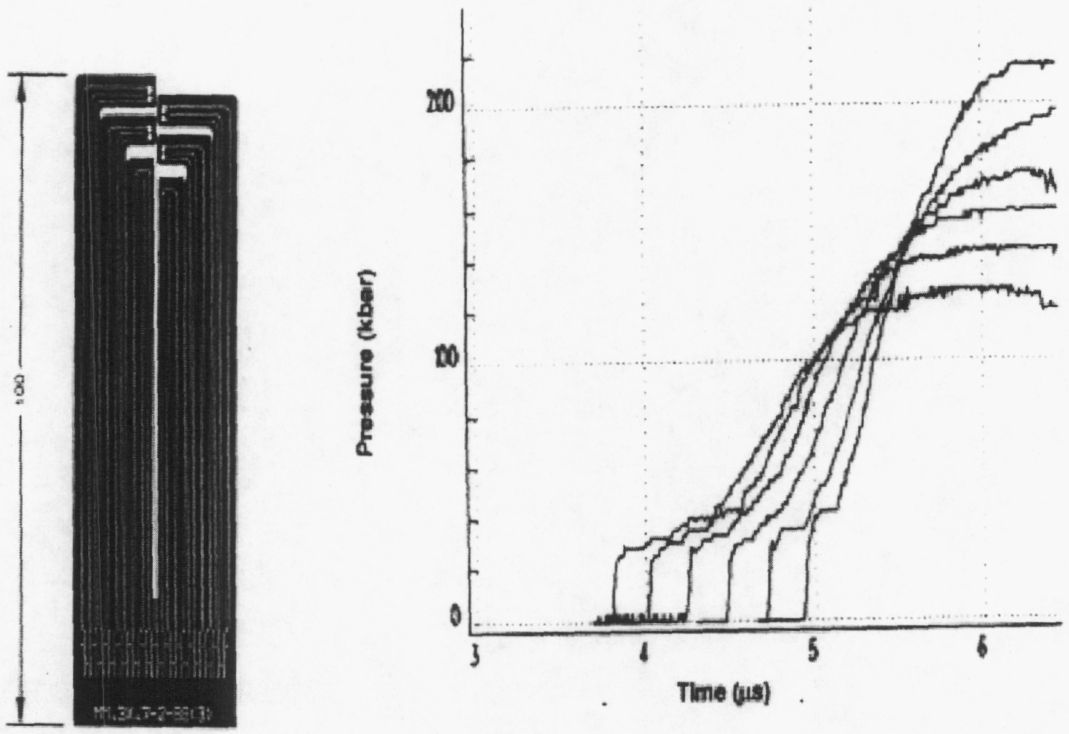

Figure 7. A six element multiple manganin foil gauge and its results in LX-14

Because the gauge elements are placed on inclined surfaces the rise time of the signal becomes of interest. In order to prevent this from becoming a detriment the width of gauge elements is kept very small i.e., 0.2 and 0.3 mm for particle velocity and manganin pressure gauges respectively. With these widths on an inclined surface of 24° a shock speed of 5 mm/μs will produce a rise time that is less than 30 ns which is quite reasonable for these types of measurements.

Other materials such as Ytterbium and Carbon are also used as pressure sensitive elements. They are usually preferred in the low-pressure regimes, below 50 kb, where manganin sensitivity is very low. However, these materials also exhibit a high sensitivity to temperature, which makes them less attractive at higher pressures where temperature changes are also significant. Their general characteristics are given in Table 1.

Table 1 . Selected additional gauges used in shock wave experiments on explosives at our laboratory

Gage name	Generally use	Nominal range of applicability	Special features	Selected references
Ytteribium foil	Low pressure 1-D Experiments	0-20 kb P ± 5-10% typically 25-115 ns temporal resolution	High piezeo-resistance coefficient giving high sensitivity; large hystersis on pressure release	19
Carbon foil	Low pressure 1-D Experiments	0-50 kb P ± 8-15% typically 25-115 ns temporal resolution	Good for use in radiation environment; peak pressure measurement; large hystersis on pressure release	23-24
Carbon resistor	Low pressure 1-D and 2-D experiments	0-50 kb P ± 8-15% typically 1.4-1.5 μs temporal resolution	Very durable and can last for milliseconds ; used in difficult environments such as large grain materials; good peak pressure measurement; large hystersis on pressure release	25-27
PVDF foil	Medium pressures for 1-D experiments	0-100 kb {some claim up to 300 kb] P ± 5-10% typically 25-115 ns temporal resolution	Very fast response pizeo-electric gauge, which requires no power supply for use. Current mode recording gives pressure derivatives directly; very sensitive to lateral strain	28-31

PVDF PRESSURE GAUGE

Another type of dynamic pressure measuring device is the PVDF (Polyvinylidene Fluoride) gauge, which is still in its development stage. Its primary advantages are in its simplicity of operation. In contrast to the just-described foil gauges it does not require an energy source, such as a constant current power supply for the manganin gauge, or magnet for the particle velocity gauge. It has its own stored energy in the form of its remnant polarization. The polyvinylidene fluoride is a semicrystalline polymer whose monomer is CH_2-CF_2. It is approximately 50% crystalline and 50% amorphous. In 1969 Kawai[32] found that this material becomes strongly piezoelectric when it is first subjected to a mechanical stretching and subsequently to a slow cycling by a strong electrical field. An attempt to use this material as a pressure-measuring element was first described by Bauer[33] who has since developed the gauge for commercial use. A thorough analysis of the gauge was given at a workshop conducted at Sandia Laboratories in 1991[34]. Considerable amount of work on these gauges was also done by Charest and his associates[31]. At the Lawrence Livermore National Laboratory we are using these gauges in field applications where simplicity is considered a major advantage.

SUMMARY

The diagnostic techniques just described form the basic arsenal of experimental tools for the high-pressure work on reactive and nonreactive materials. The study of time dependent flow has become more important for developing constitutive relations for shocked materials, time dependent rate data on phase transitions and chemical rections especially now with advances in kinetic theories and gauges offering spatial and temporal resolution capable to measure these phenomena. It was

also shown how new and innovative physical principles find their way into an experimental laboratory such as a ferroelectric property of a polymer turns a rather unsophisticated plastic material into a simple but reliable pressure gauge.

The ever-increasing capability in the computer technology leading to more sophisticated numerical modeling of physical phenomena puts significant pressure on the experimental accuracy of our measurements. The demand is not only for more accurate measurement during the event but also for a longer time after the event. This puts the additional constraints on the diagnostics that they must be durable and reliable. Such addition of constraints will never stop and will continue with much stronger demands. That makes our effort of upgrading and improving of the experimental diagnostic an important and continuous process.

In addition with the advances in computational capabilities there is an increased need to develop techniques to measure two and three-dimensional flow parameters has arisen. Readers are directed to two papers in this conference by these authors on two-dimensional gauging.

ACKNOWLEDGMENTS

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